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COMPATIBILITY OF POLYMERIC FILMS WITH LIQUID OXYGEN

by

J. T. Hoggatt

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INTERIM REPORT

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John T. Hoggatt

Prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

January 1967

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Technical Management
NASA Lewis Research Center
Cleveland, Ohio
Liquid Rocket Technology Branch
Chemical Rocket Division
R. F. Lark

THE BOEING COMPANY
Aerospace Group
Seattle, Washington

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ABSTRACT

Liquid oxygen impact sensitivity levels were determined for single and multi-ply Mylar, Kapton and FEP Teflon coated Kapton polymeric film samples. The effects of stacking; the addition of a TFE Teflon fabric cushioning ply; and long term LOX exposure were determined. Tests were conducted per the Army Ballistic Missile Agency (ABMA) impact test specification.

FOREWORD

This report was prepared by The Boeing Company, Space Division, under NASA Contract NAS 3-7952. The work was administered by the Lewis Research Center, Liquid Rocket Technology Branch, Chemical Rocket Division, Cleveland, Ohio with Mr. Raymond F. Lark as program manager.

Contract NAS 3-7952, "Development of Liquid Oxygen Positive Expulsion Bladders" is an 18-month program (July 1966 to January 1968) consisting of three separate but related tasks. This interim report covers the work performed under Task I (July 1966 to January 1967) of that program.

Performance of this contract is under the direction of the Materials and Processes organization, Spacecraft Mechanics and Materials Technology, Space Division of The Boeing Company. Mr. C.D. Burns is Program Supervisor and Mr. J.T. Hoggatt Program Manager and Principal Investigator. Liquid oxygen impact testing was conducted by Mr. M. Dickinson. Contributions by Mr. P.B. Kennedy and Mr. A.D. VonVolkli to the program and in the preparation of this report are gratefully acknowledged.

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The Boeing Company

1.0 SUMMARY

This program represents the first task of a program to develop a polymeric positive expulsion bladder for liquid oxygen propellant systems. The prime objective of Task I was to select polymeric film materials and material configurations for future bladder fabrication. To aid in making a selection, the liquid oxygen impact energy threshold values of twenty different polymeric sample configurations were determined. These samples consisted of four basic polymeric films; 1/4-mil and 1/2-mil Mylar, 1/2-mil Kapton and 1/2-mil Kapton film coated both sides with 1/2-mil FEP Teflon, in single and multi-ply configurations. TFE Teflon fabric was placed between the film plies of select samples to investigate the possible decrease of the laminate impact sensitivity by increasing its energy absorbing capabilities. Each ply of the sample, including the fabric, contained a butt seam with an adhesive bonded reinforcing doubler on each side. The effects of a multi-ply sample, the influence of Teflon fabric as an energy absorbing media, and the effects of long term liquid oxygen exposure on the impact sensitivity level of a given material were determined. The impact sensitivity tests were conducted per the standard ABMA specification pertaining thereto. This test method was determined to be the most reliable test for initial material screening. Final testing of materials for LOX compatibility will be accomplished in subsequent program tasks by actual expulsion cycling the bladders, in the presence of LOX, under severe static and dynamic conditions.

It was shown that the ABMA test method for impact sensitivity determinations, which was designed for single-ply materials, could be adapted for multi-ply sample testing. However the results between multi-ply and single-ply tests do not appear

to have a direct correlation. Multi-ply testing of a given material results in a greater "indicated" LOX impact sensitivity than does the same material in single-ply form. Additional testing with a wider variety of materials is required to establish a limit of acceptability for multi-ply samples.

None of the twenty laminated configurations tested passed the ABMA impact acceptability requirement of 72 ft-lb, even though in some samples all the individual material components are rated as LOX compatible. In subsequent tests it was verified that single plies of plain Kapton film, FEP coated Kapton film and TFE Teflon fabric are LOX compatible.

The effects of long term LOX exposure of polymeric materials upon their impact sensitivity was established by submerging selected laminated samples in LOX for a period of 14 days and then testing. The exposure resulted in both a greater reactivity and a greater sensitivity to impact.

Mylar and Kapton film, without substrate plies, were selected as materials for bladder fabrication.

2.0 INTRODUCTION

The ABMA* LOX impact sensitivity test was established to provide a much needed standard of comparison for determining a given material's sensitivity to ignition resulting from impact in a LOX environment. (References 1-4). Under this standard, a material that fails to char, flash or explode under an impact of 72 ft-lbs of energy in twenty consecutive test determinations is considered LOX compatible, enabling it to be used in LOX systems. Polymeric materials have found only limited use in liquid oxygen environmental applications, such as in propulsion systems, due to their relatively high LOX impact sensitivity under this test. Consequently, the probability of developing a polymeric film combination that was both flexible at LOX temperatures and fully LOX compatible according to ABMA specifications was considered somewhat remote. Nevertheless, the ABMA test method was considered to be the most reliable test for initial material screening even though the 72 ft-lb requirement was considered high for expulsion bladder materials. Final testing of the materials for determination of LOX compatibility will be conducted by cycling bladders under rigid static and dynamic expulsion tests in a LOX environment. The results of the bladder tests will then be compared to those obtained by the ABMA test.

Extensive data is available on a multitude of polymeric materials, all of which were tested in a single ply form in accordance with the ABMA test requirements. Unfortunately most polymeric materials, especially films, are not used in the "as-received" condition but are usually bonded or laminated together or to another substrate material. The current ABMA test has not been extended to the testing of thick or multi-ply sample testing. The established limit of acceptability (72 ft-lb) does not reflect in any way the intended use of the material nor does it account for the fact that two supposedly compatible materials when laminated or used in multi-layer construction may not be acceptable if retested in these configurations. This inconsistency arises from voids and stress concentration developed in the processing of the materials. These discontinuities can be

*Army Ballistic Missile Agency, Huntsville, Alabama.

sufficient to lower the impact sensitivity level of the composite below acceptable limits. Impact data is needed that reflects these influences. The limit of acceptability should in turn take into account the degree of impact the material may receive in service. In some instances this may exceed 72 ft-lb of energy and in others, under the most severe conditions, it may be substantially less.

Therefore, four flexible polymeric films of known LOX impact sensitivity were selected to investigate the effect of certain processing and usage parameters upon the relative LOX impact sensitivity levels of the materials. The parameters were chosen with the materials intended use in the construction of LOX positive expulsion bladders. These were: (1) the effects of an adhesive joint, (2) the influence and practicality of multi-ply sample testing, (3) the influence of the addition of a shock damping material, and (4) the effects of long-term LOX exposure.

The material combinations tested which showed the highest impact-insensitivity values were selected for the bladder fabrication and LOX testing under subsequent program tasks.

3.0 TEST PROGRAM

3.1 MATERIAL SELECTION

The material selection for this program was limited to those shown in Table I and was based solely on impending usage in expulsion bladder applications. Mylar, Kapton and FEP Teflon-coated Kapton were selected as the basic barrier film materials; and TFE Teflon fabric as the basic substrate and abrasion ply material. Mylar, even though known to be LOX impact sensitive, was chosen for its demonstrated performance as a cryogenic bladder material (References 5, 6 and 7). The Kapton film was selected principally for its excellent flexibility at -320°F, its high threshold of impact insensitivity (approximately 72 ft-lbs) and demonstrated performance as a bladder material (References 7, 8, and 10). The third material, Kapton film with Teflon film laminated to each surface, was chosen for its LOX impact insensitivity qualities. Although the flexibility of the coated film is known to be lower than that of pure Kapton film at cryogenic temperatures (Reference 10), it is still considered acceptable, and the material is LOX impact insensitive by ABMA standards (Reference 9).

For the abrasion and substrate ply material, bleached TFE Teflon fabric was selected. This material is LOX impact insensitive at 72 ft-lbs and has good flexibility and cyclic endurance at cryogenic temperatures. It was originally intended to use TFE felt in place of the fabric; however, the minimum commercially available thickness was 1/16", which is too heavy for practical bladder usage.

The adhesive selection for the program was a thermoplastic polyester adhesive, GT-300. The adhesive is LOX impact sensitive and is considered a poor choice from that standpoint. On the other hand, most available adhesives are impact sensitive, and the few that are not sensitive either do not lend themselves to

TABLE I - MATERIAL SELECTION

Material Description	Chemical Composition	Thickness (mils)	Vendor Designation	Supplier
Mylar Film	Polyester	0.25	Type C	E.I. duPont de Nemours Co.
Mylar Film	Polyester	0.50	Type C	E.I. duPont de Nemours Co.
Kapton Film	Polyimide	0.50	50XH755	E.I. duPont de Nemours Co.
TFE Fabric	Fluorocarbon	3.0	T-138	Stern and Stern Textiles, Inc.
1/2-mil Polyester Adhesive on 1/2-mil Mylar Film	Polyester	1.0	GT-300	G.T. Schjeldahl Co.
1/2-mil Kapton Coated Both Sides with 1/2-mil FEP Teflon	Polyimide/ Fluorocarbon	1.5	150XHF999C	E.I. duPont de Nemours Co.
1/2-mil Kapton Coated One Side with 1/2-mil FEP Teflon	Polyimide/ Fluorocarbon	1.0	100XHF099C	E.I. duPont de Nemours Co.

acceptable bladder fabrication techniques or do not give satisfactory performance at cryogenic temperature. Therefore, for the lack of a better system, GT-300 was used. FEP Teflon was used whenever possible as a thermoplastic adhesive (sealing medium). This material could only be used to seal FEP-coated Kapton to itself or to the TFE fabric since FEP Teflon does not adequately seal to plain Kapton or Mylar film.

3.2 SPECIMEN PREPARATION

The individual test specimens consisted of one of three basic barrier film materials, Mylar, Kapton, or FEP/Kapton/FEP, plies with and without Teflon fabric, in the sequence shown in Table II. Each ply of the sample contained one of the five seam configurations illustrated in Figure 1 and described below, with no bond or seal between plies. Shown in Figure 2 is a typical test specimen.

The individual seams were fabricated as follows:

A. Mylar Seam (Figure 1-A)

This seam was used for both the 1/4-mil and 1/2-mil Mylar barrier plies. In each instance, the 1" wide film strips were butt jointed and bonded on both sides with GT-300 polyester adhesive. The bonding operation was accomplished using a hand sealing iron set at 350° ±20°F. Each side of the film was sealed separately.

B. Kapton Seam (Figure 1-B)

The procedures for making the Kapton seam were identical to those utilized for the Mylar films (item A above) with the following exception:

Prior to seam fabrication all of the Kapton film was heat treated to remove traces of residual solvent by heat aging the film at 450° ±10°F for a period of 48 hours. Data has indicated that Kapton film is LOX compatible

TABLE II - IMPACT TEST SPECIMEN CONFIGURATIONS

Specimen Configuration	Barrier Film	No. of Barrier Film Plies	No. of Substrate Plies	Abrasion Ply [1]	Total No. of Plies/Sample
1	1/4-mil Mylar-C Film	1	0	No	1
2		10	0	Yes	12
3		20	0	Yes	22
4		10	11	Yes	23
5	1/4-mil Mylar-C Film	20	21	Yes	43
6	1/2-mil Mylar-C Film	1	0	No	1
7		10	0	Yes	12
8		20	0	Yes	22
9		10	11	Yes	23
10	1/2-mil Mylar-C Film	20	21	Yes	43
11	1/2-mil Kapton Film	1	0	No	1
12		10	0	Yes	12
13		20	0	Yes	22
14		10	11	Yes	23
15	1/2-mil Kapton Film	20	21	Yes	43
16	1/2-mil Kapton with 1/2-mil FEP both sides.	1	0	No	1
17		10	0	Yes	12
18		20	0	Yes	22
19		10	11	Yes	23
20	1/2-mil Kapton with 1/2-mil FEP both sides.	20	21	Yes	43

[1] Includes an abrasion ply on both top and bottom of sample.

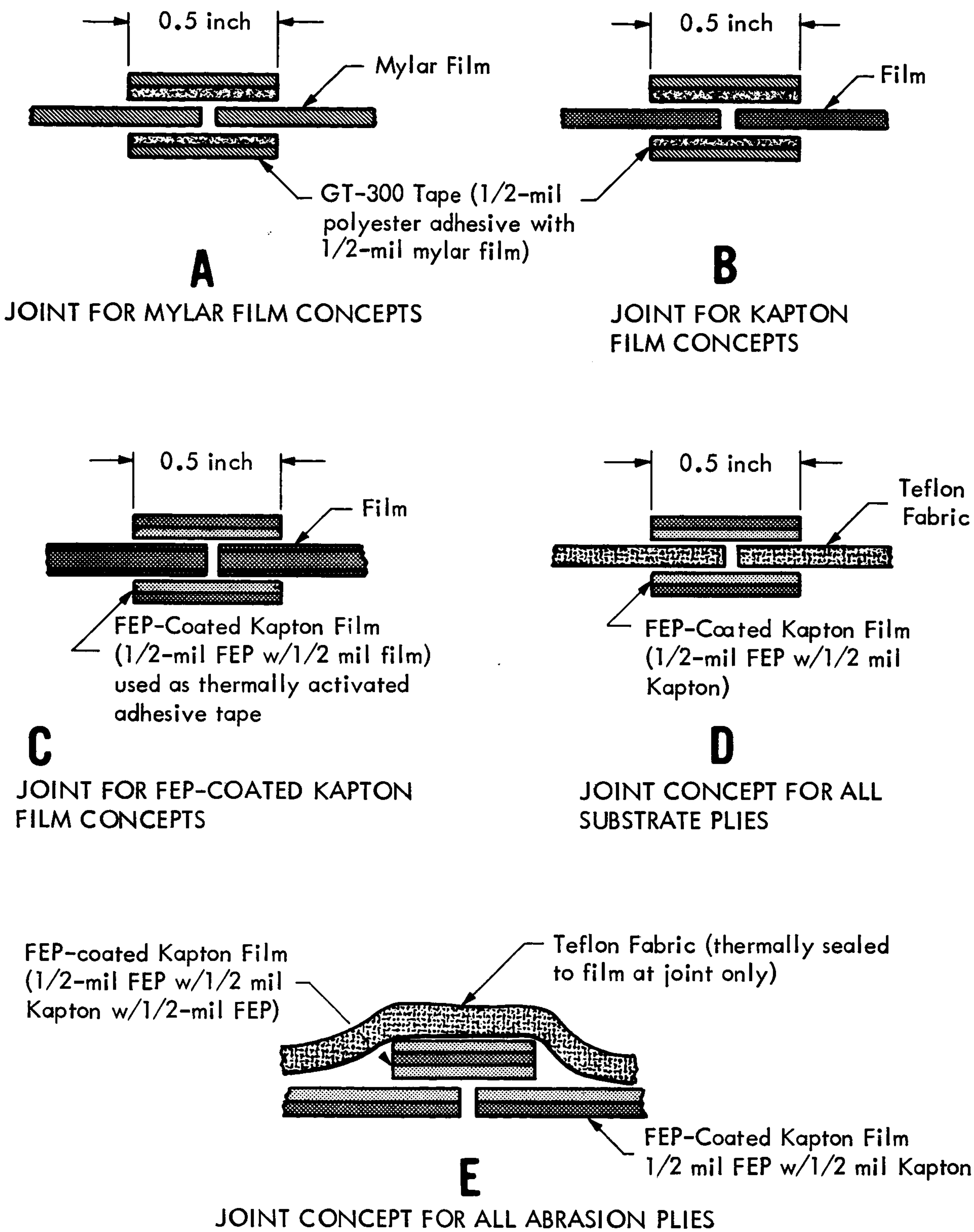

FIGURE 1 - JOINT CONCEPTS

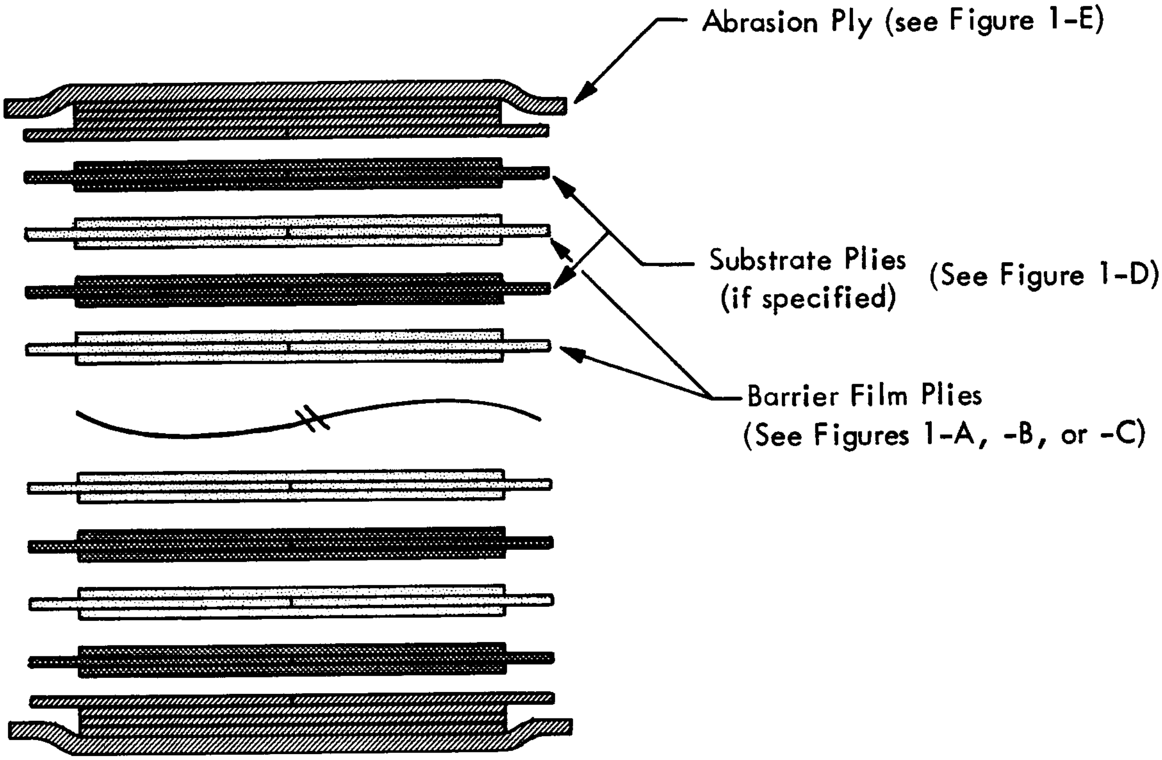

FIGURE 2 - REPRESENTATIVE TEST SPECIMEN - LOX IMPACT TEST

(References 2 and 9) if the residual solvent content is below 0.3%. Normally the residual solvent content of "as received" material ranges from 0.2-0.5%. Due to this residual solvent variation, Kapton is given a "batch" rating (Reference 2); that is, some lots are LOX compatible while others are not, and each lot must be tested individually.

With the use of thermogravimetric analysis techniques it was determined that 48 hours at 450°F was sufficient to lower the residual solvent content of the purchased film to the acceptable 0.3% limit. LOX impact tests (Table IV) were conducted on the film to verify that this treatment was satisfactory.

It should be noted that higher treating temperatures could be used, which would result in shorter exposure times. However, it was desirable to use the same exposure cycle for both the FEP/Kapton and plain Kapton film. Because the FEP Teflon imposes temperature limitations ($<$ 525°F), the temperature of 450°-475°F was selected. The FEP/Kapton/FEP film was not heat treated since it has been given a satisfactory rating under "as-received" conditions.

C. FEP/Kapton/FEP Seams (Figure 1-C)

The coated Kapton film was prepared in the same manner as the two previously mentioned barrier film plies, except in place of the GT-300 adhesive, FEP/Kapton was utilized as the adhesive. The 1/2" wide FEP/Kapton film was heat sealed to the FEP/Kapton/FEP film using a sealing iron set at 615° ±10°F. The film was applied to each side of the barrier film in separate sealing operations. The FEP/Kapton was heat treated prior to use (see Item B. above for treating process).

D. Substrate Seams (Figure 1-D)

TFE Teflon fabric was used for all substrate plies. The fabric was joined using a butt joint and reinforcing doublers on each surface as shown in the illustration. The doubler, which consisted of Kapton/FEP film, was heat sealed to the fabric. An iron temperature of 615° ±10°F was used. Each doubler was sealed to the fabric in separate operations.

The TFE fabric used for both the substrate plies and the abrasion plies was bleached and dimensionally stabilized prior to use, using the following process:

> Loosely folded fabric was placed in a clean vented oven for 4 hours at 475°F followed by 48 hours at 575°F. A 22-25% area reduction occurred during the conditioning process due to shrinkage of the fibers; however, a closely-woven, dimensionally stable white Teflon fabric resulted. The treating process is required to obtain a LOX compatible material (Reference 2).

E. Abrasion Seams (Figure 1-E)

As shown in Figure 1-E, the abrasion ply consisted of FEP/Kapton joined to TFE Teflon fabric with FEP/Kapton/FEP film. Again this seam was fabricated using a two-step hand process. In the first step the FEP/Kapton barrier film was joined together with the FEP/Kapton/FEP. In the second step, the TFE fabric was sealed to the film laminate.

Each of the seams mentioned above was fabricated under clean room conditions. The materials were thoroughly cleaned with methyl ethyl ketone and dried prior to use to remove fingerprints, foreign particles and other contaminants which could be entrapped during the seam fabrication. Nylon cloth was used to clean the materials to prevent the entrappment of lint. Figures 3 and 4 show the cutting and sealing tables used.

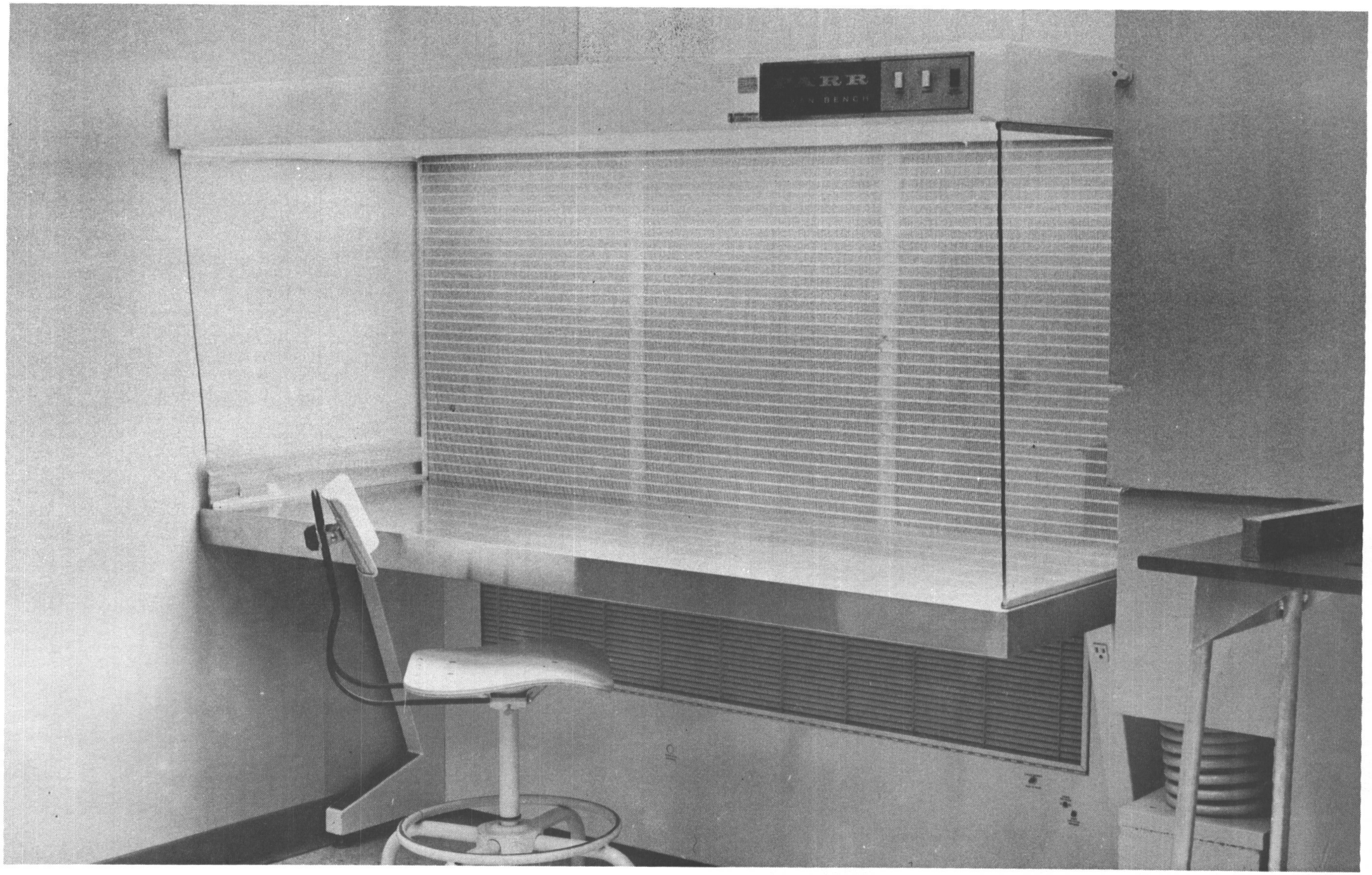

FIGURE 3 - LAMINAR FLOW CLEAN BENCH

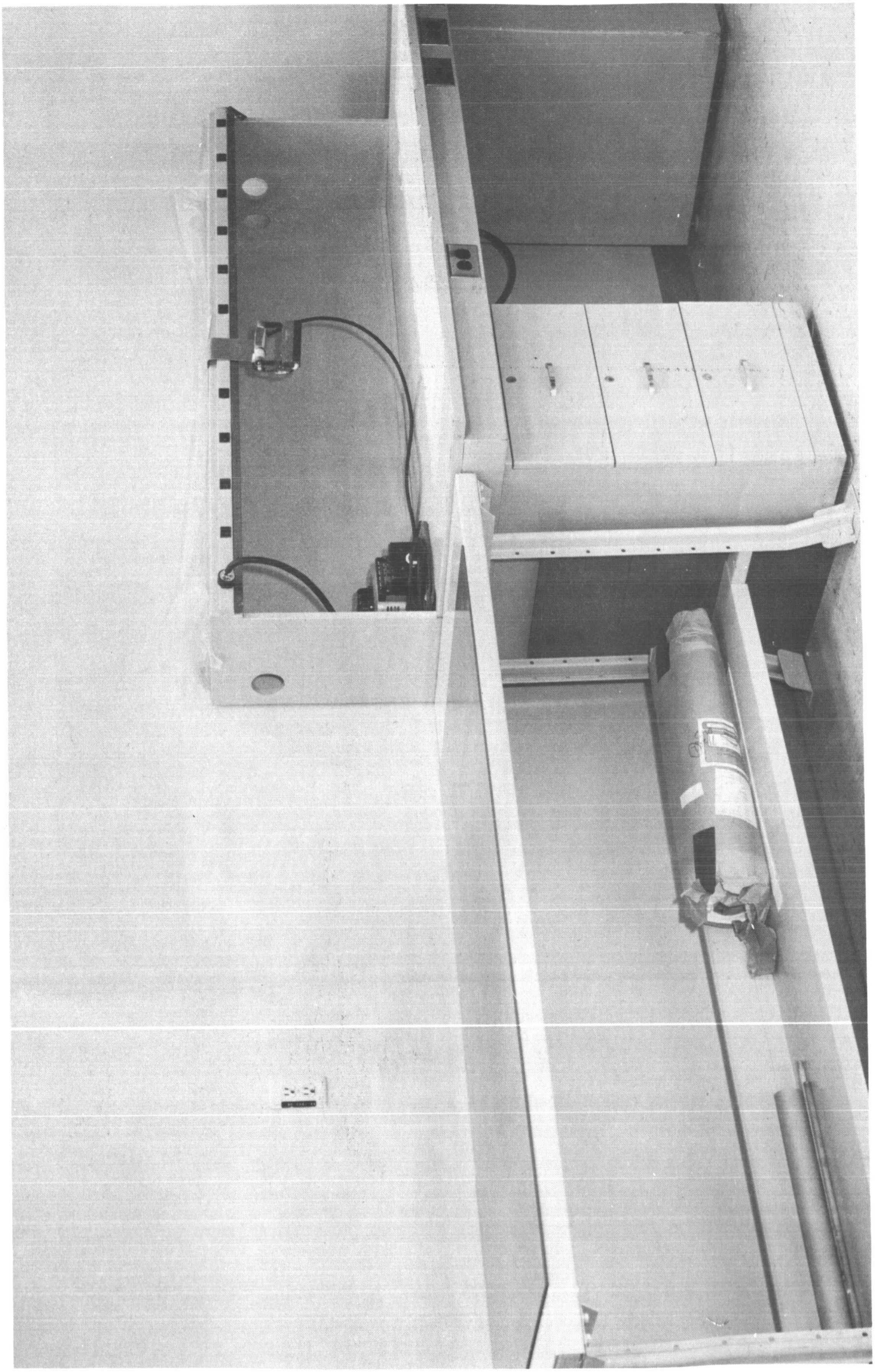

FIGURE 4 – CUTTING TABLE AND SEALING BENCH

Following completion of the seams, the film strips (approximately 2" wide x 6 feet long) were transported to the Boeing Tulalip Test Site where the LOX impact testing was accomplished. This facility is equipped with a "preparation room" especially designed for support of LOX impact testing. In this facility each film strip was washed in a hot detergent (Diversey 909) solution, scrubbed and then rinsed in tap water, followed by a rinse in deionized water. Each strip was then dried at 180-212°F in a stainless steel conditioning oven for 30-60 minutes.

Individual film discs, 11/16" in diameter, were stamped from the clean strips directly over the seam area. A Teflon pad was used in the stamping operation to prevent detrimental contamination. The plies or discs were then assembled in the sequence prescribed in Table II. With the exception of the single-ply specimens shown in Table II, each sample contained an abrasion ply on the upper and lower surfaces. When substrate plies were required, they were spaced alternately with the barrier film plies.

Figure 5 gives a graphical presentation of the sample preparation, cleaning and assembly process.

3.3 EQUIPMENT

The LOX impact sensitivity tester shown in Figures 6, 7, and 8 was used for all impact tests stated herein. This equipment and its operation are certified to MSFC-SPEC-106A (Reference 4). A detailed description of this equipment and its operation can be found in References 1 and 3; however, shown in Figures 9 and 10 are detailed schematics of the anvil, striker pin and sample cup.

In the ABMA LOX impact test a plummet of known mass is dropped, under near-frictionless conditions, from a given height, impacting a small test sample contained in an aluminum cup and submerged in LOX (see Figure 9). If the material is LOX impact sensitive it will char, flash or explode. For a

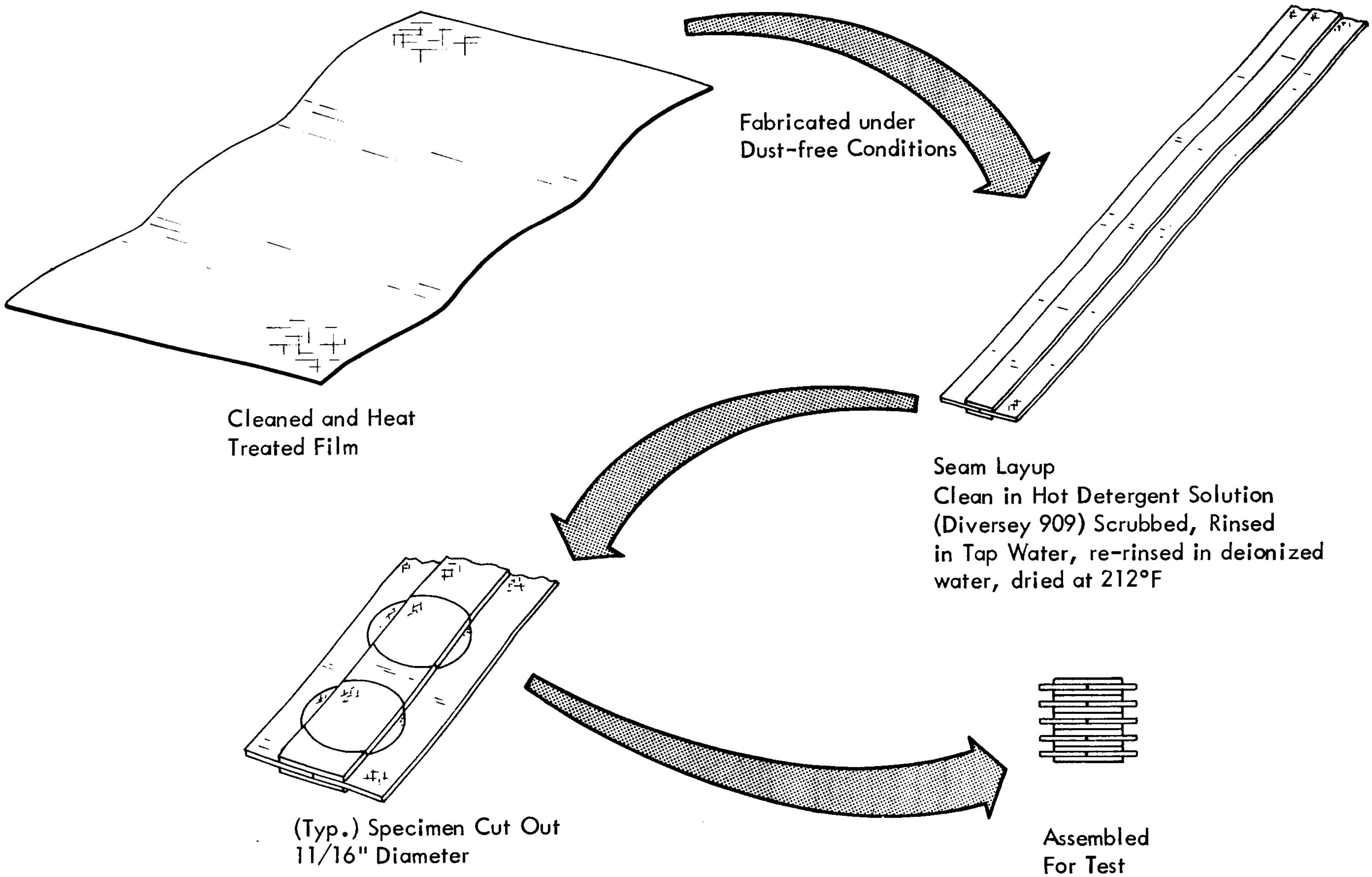

FIGURE 5 - FABRICATION SEQUENCE - LOX IMPACT SENSITIVITY SPECIMENS

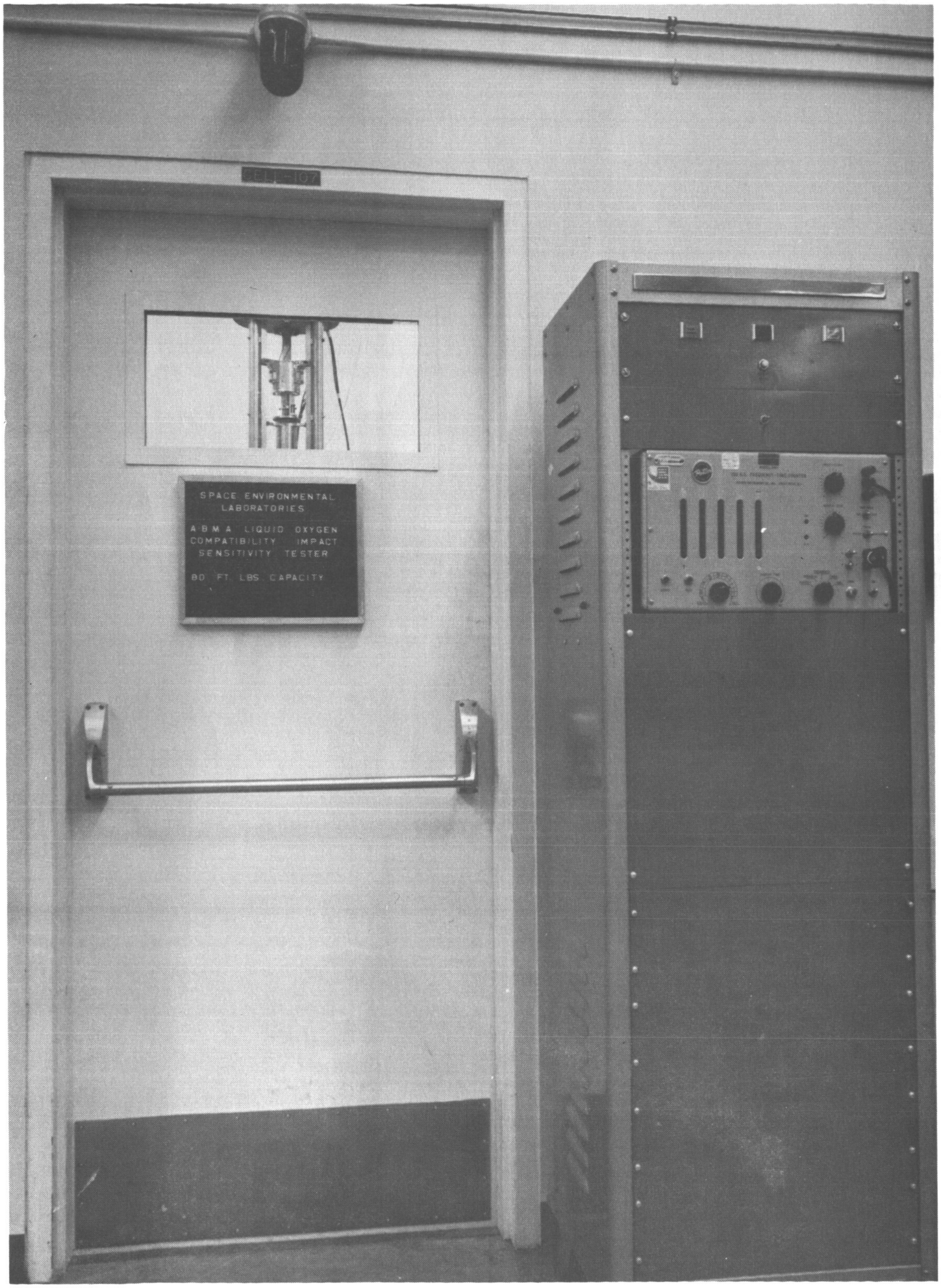

FIGURE 6 - TEST CELL AND CONTROL CONSOLE

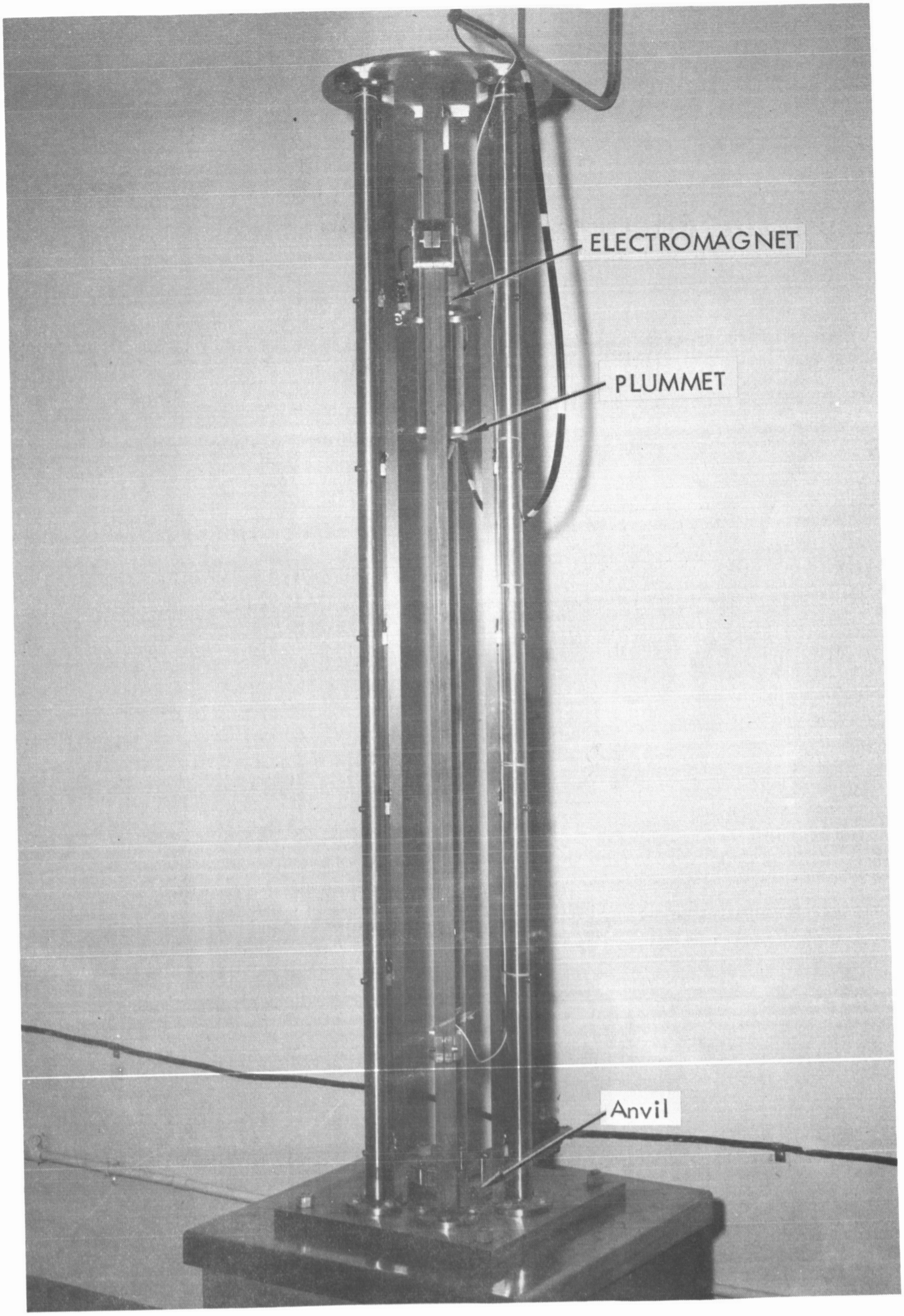

FIGURE 7 - TEST EQUIPMENT - LOX IMPACT SENSITIVITY

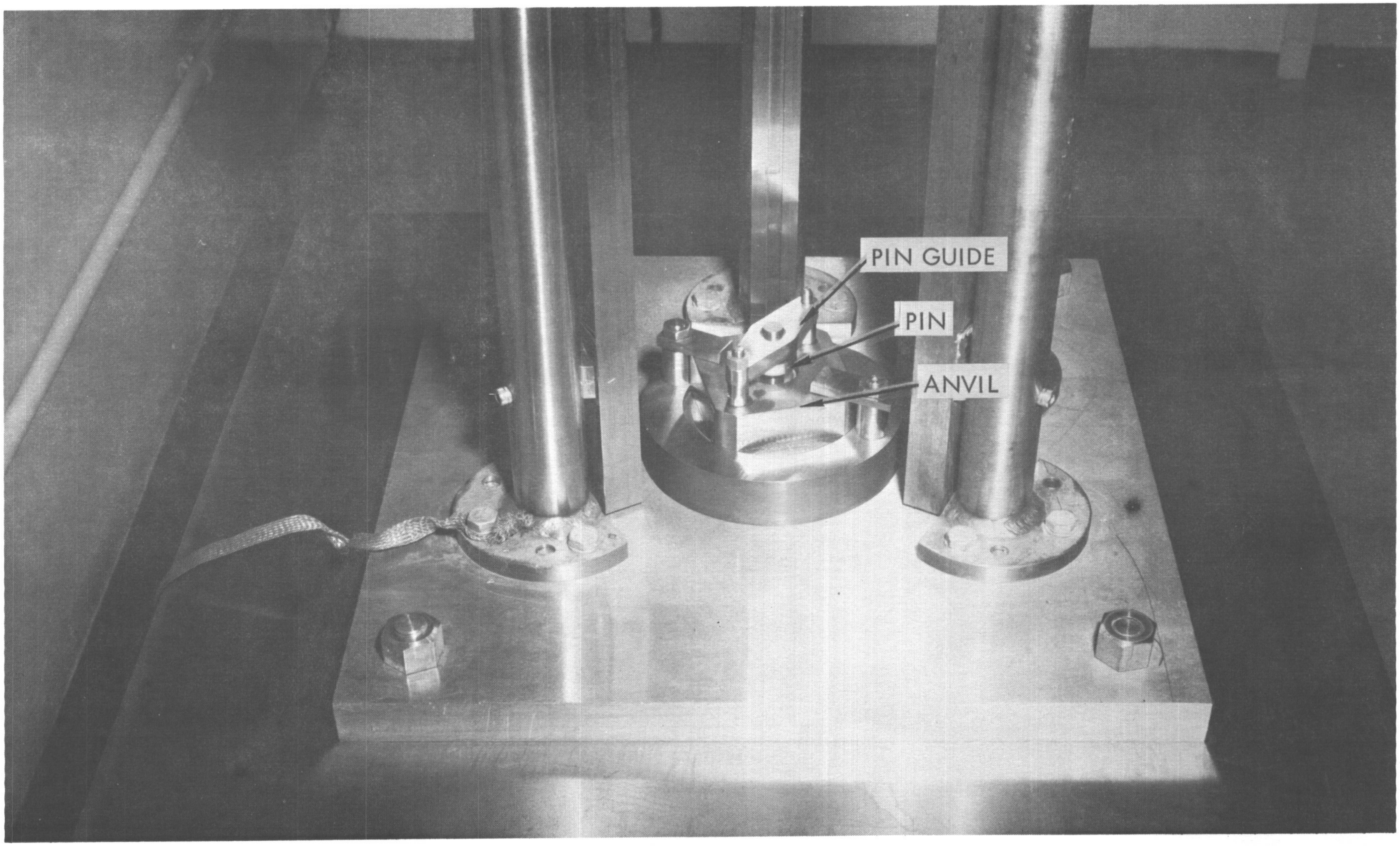

FIGURE 8 - TEST EQUIPMENT - ANVIL

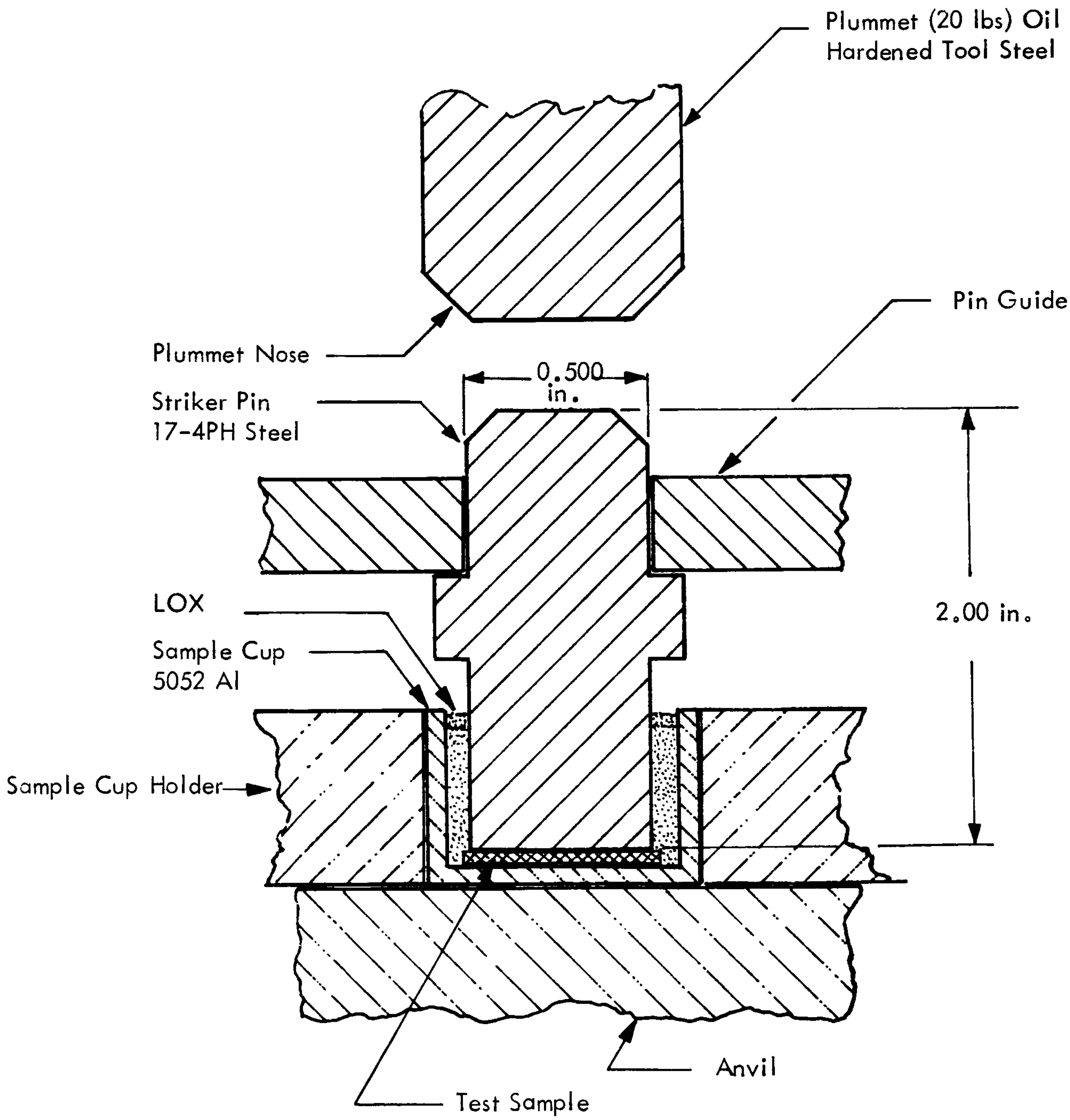

FIGURE 9 - DETAILS OF STRIKER, SAMPLE CUP, AND SAMPLE (IMPACT SENSITIVITY TESTER)

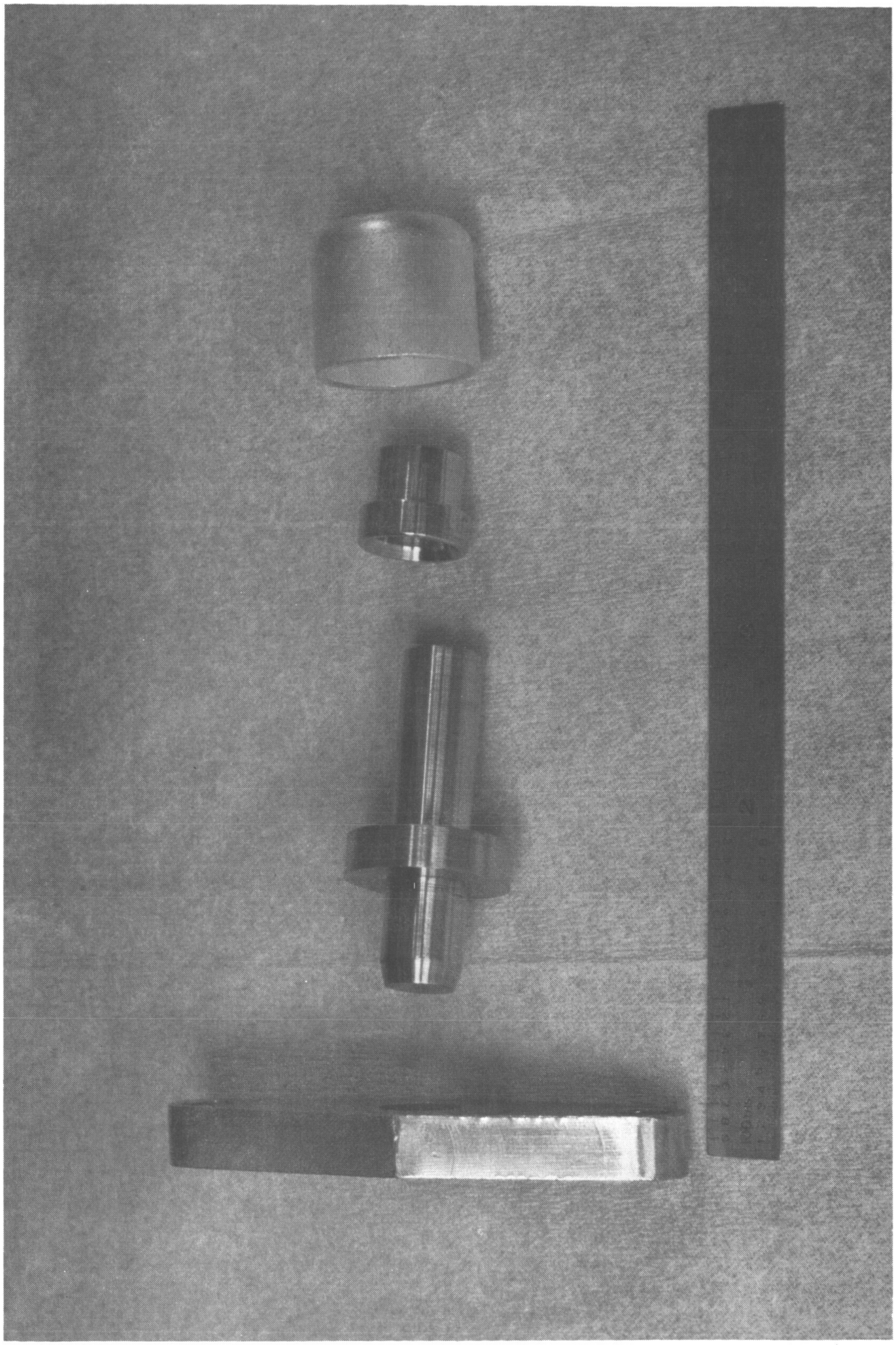

FIGURE 10 - PIN GUIDE, PIN, SLEEVE AND CUP

material to be considered LOX impact insensitive it must be capable of absorbing 72 ft-lbs of energy (9.04 kg dropped from a height of 1.1 meters) without any reaction in 20 successive trials. The threshold energy value is defined as the maximum impact energy a material can withstand in LOX without igniting (charring, flashing, etc.). Again no reactions must be obtained in 20 samples.

3.4 PROCEDURES

The equipment and support facilities were operated and maintained in accordance with the requirements of MSFC-SPEC-106A (Reference 3). The impact specimens were conditioned and tested per MSFC-SPEC-106A with the following exception:

The ABMA test was not designed to test multi-ply films or a "stack" of materials, and no provisions are made to do so; therefore, changes had to be incorporated in the test to accommodate the proposed test samples. The polymeric films used were of much lower density than LOX and had a tendency to float to the surface. In the multilayer specimens this presented a serious problem since it was extremely difficult to maintain the ply orientation and the stack alignment in the cup. As a solution, a small stainless steel sleeve was placed on the samples to keep them submerged (see Figures 11 and 13). The striker pin contacted the specimen by passing through the loosely fitting sleeve. The sleeve was light enough to permit LOX to freely circulate between the film plies yet heavy enough to restrain the film. Comparative tests were run on single ply film specimens with and without the added sleeve, and no difference in the frequency of reaction was detected.

The added sleeve provided a satisfactory means of testing all samples except those of 41 plies. On those specimens, as the striker pin impacted the sample, the lower plies were hydraulically expelled from the cup, making it impossible to determine the exact number of plies impacted. To remedy the situation a different style sleeve, shown in Figure 12, was utilized for the remaining tests.

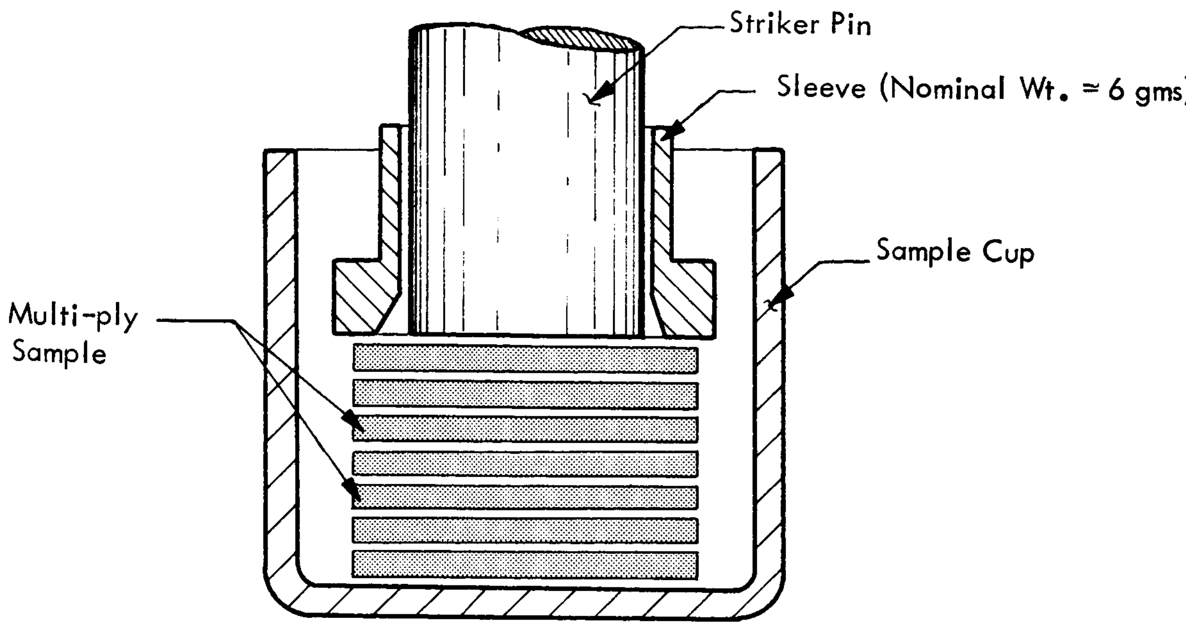

FIGURE 11

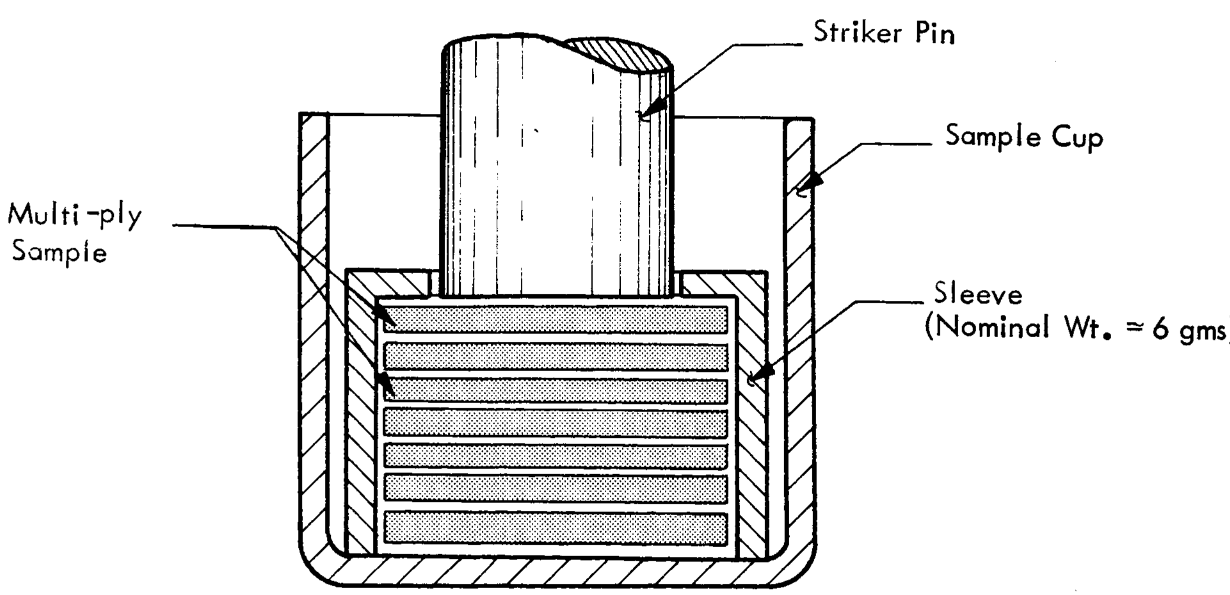

FIGURE 12

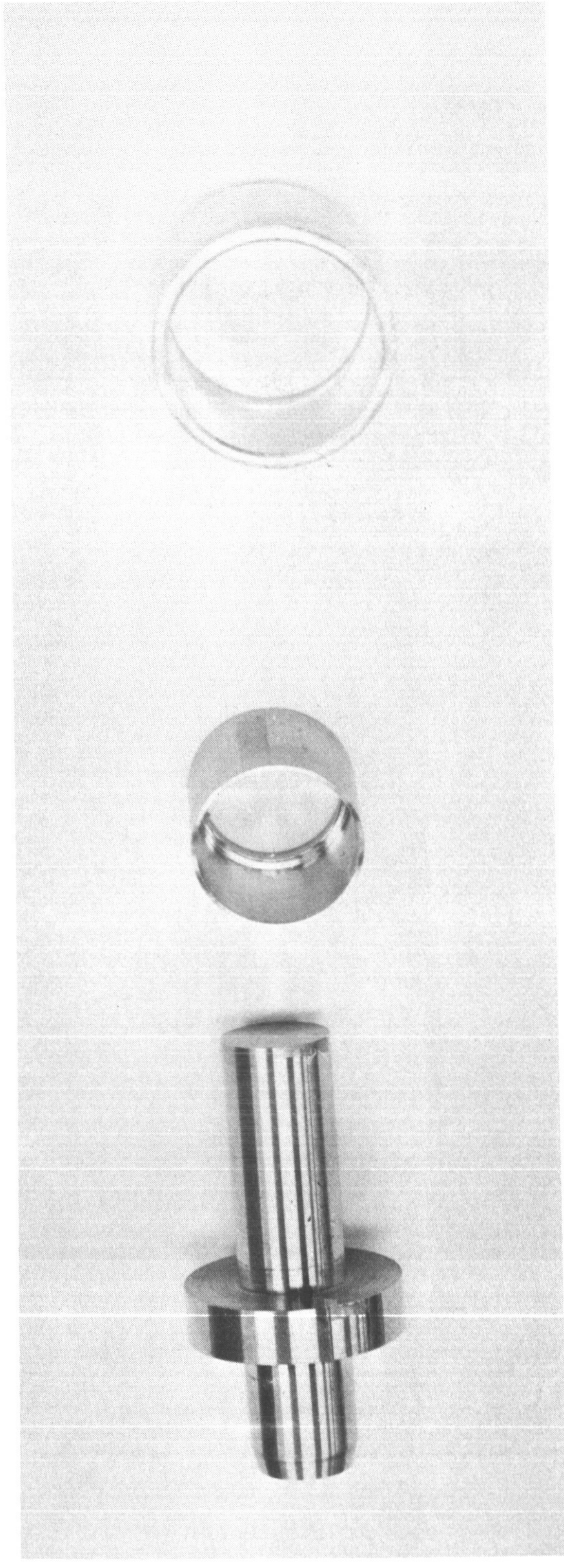

FIGURE **13** - PIN, SLEEVE AND CUP

The anvil was cooled with liquid nitrogen to minimize LOX boil-off and to assure the presence of LOX in the sample cup. The cup, sleeve, pin and sample were cooled in LOX prior to testing as required in the test specification. Figure 14 shows the LOX holding (cooling) tray.

Samples for the 14-day soak were placed in individual sample cups and weighted with a sleeve to prevent flotation. The cups were then arranged in the holding trays shown in Figure 15 and submerged in liquid oxygen for a period of 14 days. The cryostat shown in Figure 16 was used. An automatic fill device assured that a constant LOX level was maintained in the cryostat. The soak period was arbitrarily selected and intended to insure that the specimens would be LOX saturated at the time of test.

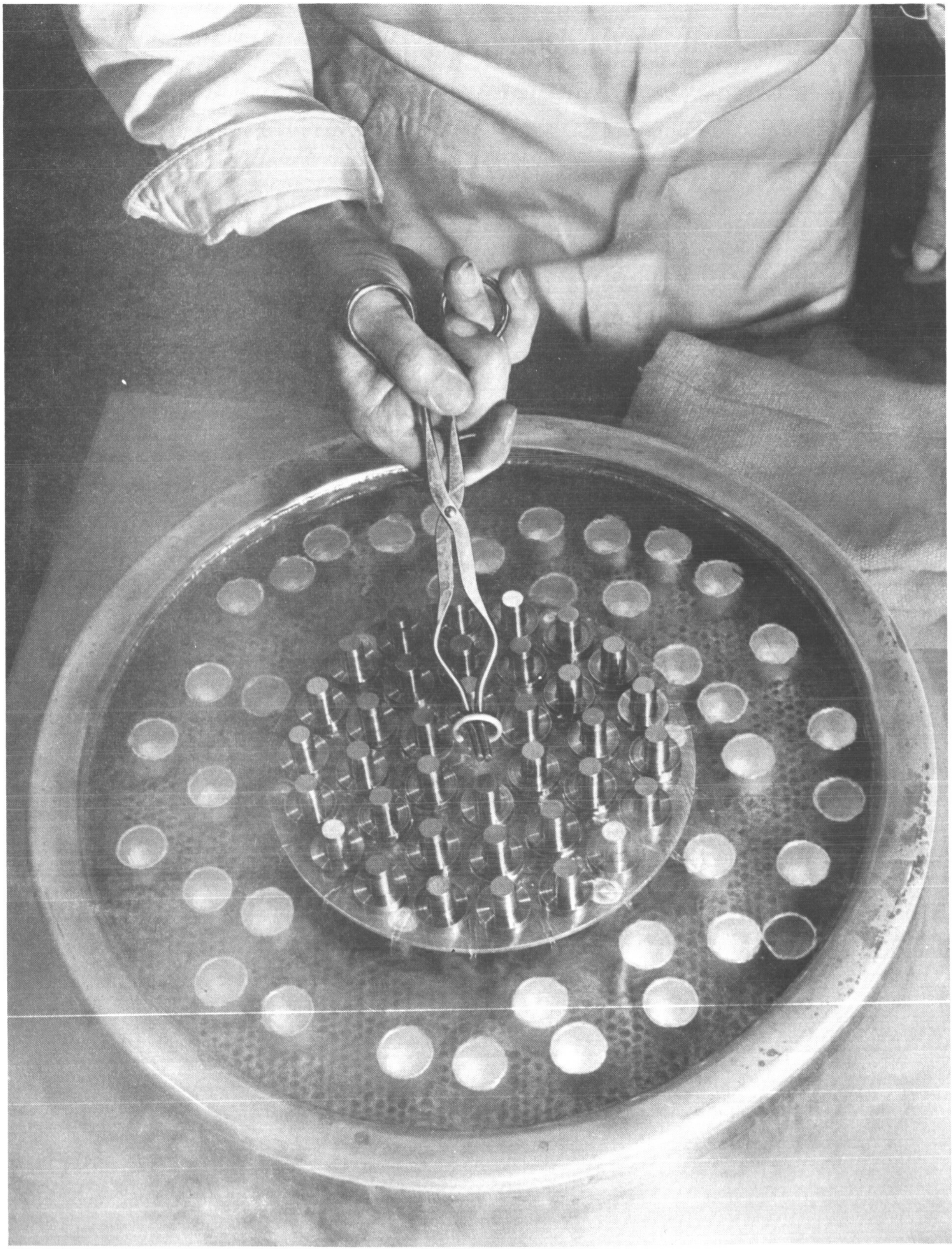

FIGURE 14 - LIQUID OXYGEN COOLING BATH

FIGURE 15 - SPECIMEN STORAGE TRAYS - 14 DAY SOAK

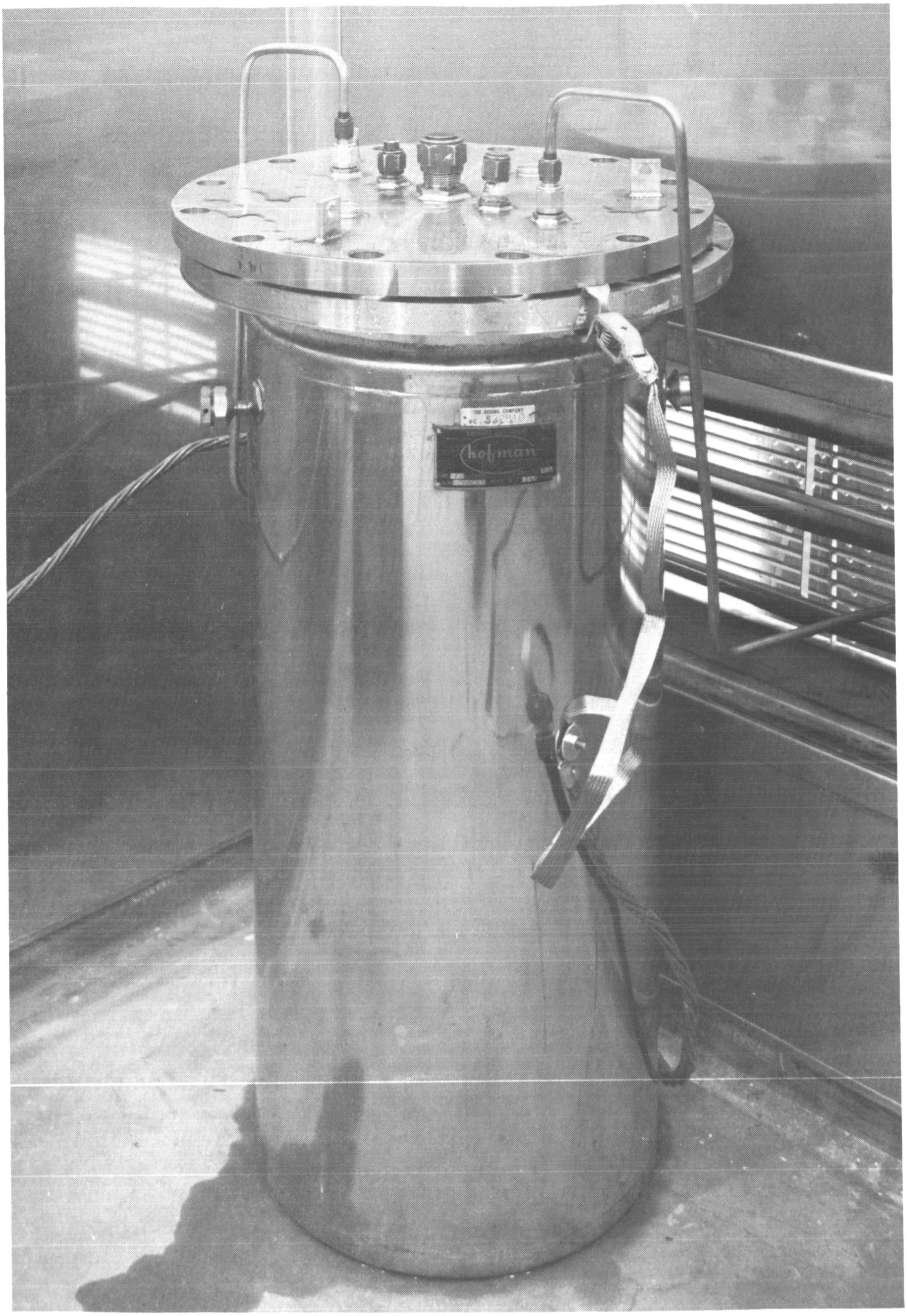

FIGURE **16** - STORAGE DEWAR - 14-DAY SOAK

4.0 RESULTS AND DISCUSSION

Tabulated in Tables III and IV are the test results obtained in this program. Table III contains data on the LOX impact sensitivity data on the single and multi-ply samples. Each ply of the samples shown in Table III contained a seam as stated in Section 3.2.

Table IV gives comparative results of plain polymeric film materials, without seams or adhesives of any nature.

Table V presents related LOX impact data for comparative purposes obtained and reported by NASA (Reference 2).

None of the film composite samples tested successfully passed the ABMA acceptance requirements of 10-Kg-m (72 ft-lbs). Of the plain materials (no seams), pure Kapton, Kapton/FEP and TFE Teflon fabric successfully passed this requirement. For each configuration, initial tests were conducted at the 72 ft-lb energy level, and then if reactions occurred, the level was lowered until the threshold value was obtained.

It is apparent from the results presented in Table III that increasing the number of barrier film plies increases the LOX impact sensitivity of the material. This is contrary to the popular belief that stacking a material would increase the threshold energy level (decrease sensitivity) due to the energy loss during compaction. Apparently during compaction (impact), the liquid oxygen which is present between each layer, and within the Teflon fabric, is expelled at an extremely high velocity and with a high shearing action across the boundary layers, causing an instantaneous temperature rise. Due to the rapid impulse-type loading, the compression is essentially an adiabatic process. Thermal conduction through the sample and to the surroundings is negligible, and temperature peaks arise. The temperature increase, although small, is sufficient to lower the apparent impact energy level required for material ignition. At the

TABLE III - RESULTS - LOX IMPACT SENSITIVITY TESTS

Configuration	Barrier Film	No. of Barrier Plies	No. of Substrate Plies	Impact Energy Level [1] - kg-m									
				10	9	8	7	6	5	4	3	2	1
1	1/4 mil Mylar	1	-	1						1	0	0	0
2	1/4 mil Mylar	10	-	10						9		13	0
3	1/4 mil Mylar	20	-	20						9		4	0
4	1/4 mil Mylar	10	11	4	8	0		0		0			
5	1/4 mil Mylar	20	21	7				8		9	3	0	
6	1/2 mil Mylar	1	-	5				3		3	1	0	
7	1/2 mil Mylar	10	-	20						20		19	15
8	1/2 mil Mylar	20	-	20						19		16	3
9	1/2 mil Mylar	10	11	17						15		8	3
10	1/2 mil Mylar	20	21	20						7		6	0
11	1/2 mil Kapton	1	-	5				4	3	0		0	
12	1/2 mil Kapton	10	-	20						20		20	9
13	1/2 mil Kapton	20	-	20						20		2	2
14	1/2 mil Kapton	10	11	20						18		8	0
15	1/2 mil Kapton	20	21	20						11	2	0	
16	1/2 mil FEP-coated Kapton	1	-	1				1	5	0			
17	"	10	-	9						9	7	0	
18	"	20	-	9						9	9	0	
19	"	10	11	5				4	7	3	0		
20	"	20	21	18				8	13	0			
1-A [2]	1/4 mil Mylar	1	-							9	6	0	
6-A	1/2 mil Mylar	1	-							9		2	0
11-A	1/2 mil Kapton	1	-					16		10		1	
16-A	1/2 mil FEP-coated Kapton	1	-					12		3		0	

[1] Number of reactions out of 20 drops.

[2] -A denotes samples submerged 14 days in LOX.

TABLE IV - RESULTS - LOX IMPACT SENSITIVITY TESTS ON PLAIN POLYMERIC MATERIALS

Material	Treatment	Impact Energy Level [3] - kg-m					
		10	8	6	4	2	1
1/4 mil Mylar	As-Received				1	0	
1/2 mil Mylar	As-Received (Lot 1)			1	0		
1/2 mil Mylar	As-Received (Lot 2)	2	0				
1/2 mil Kapton	Aged 24 hrs at 450°F	0					
1/2 mil Kapton	As-Received [2]	0					
1/2 mil FEP on 1/2 mil Kapton	As-Received	0					
1/2 mil FEP on 1/2 mil Kapton	Aged 24 hrs at 450°F	0					
TFE Fabric	Bleached	0					
Teflon-Kapton Laminate [1]	Aged 24 hrs at 450°F	0					

[1] (1/2 mil FEP-1/2 mil Kapton) film press laminated to both sides of (1/2 mil FEP-1/2 mil Kapton-1/2 mil FEP) film.

[2] Film sample supplied by NASA-LeRC.

[3] Number of reactions out of 20 test drops.

TABLE V - LOX IMPACT SENSITIVITY OF POLYMERIC MATERIALS
(Reference 2)

Material [2]	Test No. [3]	Remarks	Thickness (mils)	No. Reactions/ No. Tests	Energy Level kg-m	Rating [4]
DuPont H-Film [1]	3647		2	0/20	10	Batch test
DuPont H-Film 380-2-2	5568	Aged 24 hrs. at 300°C.	2	0/20	10	Batch test
DuPont H-Film 380-2-2	5567	No extra drying.	2	4/20	10	Unacceptable
Mylar Film	3379		6	2/22	10	Unacceptable
				2/28	5	"
				0/20	3	"
Mylar Film	3368		2	2/22	10	Unacceptable
				2/20	5	"
				0/20	3	"
Mylar Film	4545		1	4/20	10	Unacceptable
Teflon - 100X (FEP)	1247		-	0/20	10	Satisfactory

[1] Former designation for Kapton film.

[2] Manufacturer - E.I. duPont de Nemours, Co.

[3] Test numbers refer to NASA designation and data system.

[4] Refers to the ABMA rating system.

energy levels slightly above the threshold value, only slight surface charring was evident on the barrier film plies, and it had the appearance of flow rings radiating from the center of the ply. Occasional charring would also occur on the Teflon fabric substrate plies. At the 9 and 10 Kg-m level the striker pin would penetrate the upper portion of the stack, much like a hole-punch, impacting the remaining portion of the sample causing severe char damage. No charring or sign of reaction was present in the punched out areas.

It should be noted that a slight cushioning effect was detected as evidenced by the lower frequency of reaction at the lower energy levels for the multi-ply samples. This effect is dominated however by the local temperature rise effect mentioned previously.

The presence of substrate plies (Teflon fabric) between barrier film plies only slightly aided in reducing the impact sensitivity of the composite. In almost every instance, samples containing substrate plies had a threshold energy level 1 Kg-m higher than comparable samples without, but still below that of an individual barrier ply.

The GT-300 polyester adhesive and the Mylar film had approximately the same threshold energy value (3-4 Kg-m). The adhesive seemed to be the limiting factor on the Kapton film samples since the Kapton film alone was insensitive at the 72 ft-lb energy level (Table IV). It is apparent that if a LOX compatible adhesive had been available, samples could have been made that would be acceptable by ABMA standards.

Intuitively, it would be expected that configurations 16 through 20 (Table III) would pass the 10 Kg-m requirements since they are composed entirely of impact insensitive materials. Unfortunately this was not the case. The reactions indicated in Table II for configurations 16 through 20 consisted of very slight chars, and appeared to be due to the shearing phenomenon previously stated. In testing configuration #16, few reactions occurred, but the few that did were in the seam lamination. It seems that during the heat sealing process, small occasional voids or pockets would form. Upon impact, localized hot spots would result from the adiabatic compression of the trapped gas bubbles; causing subsequent charring. To verify this, similar samples were press laminated to eliminate the possibility of voids and tested at 72 ft-lbs. No reactions occurred as shown in Table IV, indicating the importance of void-free seams. This phenomenon has been experienced previously by other investigators (Reference 11). The variation in the FEP film thickness resulting from the sealing operation is another possible "triggering" mechanism (Reference 12). Slight discontinuities and voids in the Teflon adhesive layer interrupt the smooth transfer of energy through the sample. As a result, small quantities of impact energy and changes in internal bond energy are transferred as increased vibrational energy or thermal energy (temperature peaks) on a localized basis to the boundary layer atoms. This energy transfer, in conjunction with the pressure shock waves and the natural frequencies in the system, can cause "localized hot spots" or ignition. With standard hand sealing operations a few discontinuities are to be expected since it is extremely difficult to eliminate minute pockets and slight flow ridges, especially with a material with flow characteristics like FEP Teflon. In the multi-ply configurations (17 through 20), it is difficult to say to what extent the voids contributed to the low threshold values and the extent that can be contributed to the stacking effect. From the high degree of surface charring found and the increased frequency of reaction, it is believed that the stacking is the predominating factor.

It should be noted that all reactions obtained, with the exception of those from the soaked specimens, consisted of charring and in most instances a flash. In no test did an audible report occur. With the multi-ply samples it was necessary to screen the sample after test since charring would occur at random and at times no flash would be evident. The degree of charring would range from minute or surface discoloration to complete sample destruction as shown in Figure 17. A specimen was considered sensitive if noticeable charring was present anywhere within the specimen. Figure 18 shows resulting damage to a cup and pin after a severe reaction.

Comparing the data in Tables III and IV, it appears that configurations 16 through 20 are basically LOX insensitive at the 10 Kg-m energy level; however, the test method and specimen fabrication produce side effects which increase the sample sensitivity.

14-Day Soak

The polymeric film samples exposed to LOX for a period of 14 days were very reactive and had a lower threshold value than the equilvalent unsoaked samples. The samples reacted violently with a flash and loud report, as compared to a mere flash with the unsoaked materials. This indicates that a considerable amount of LOX is absorbed by the samples. At the moment of impact a greater portion of the sample is in contact with the LOX, resulting in a greater reaction.

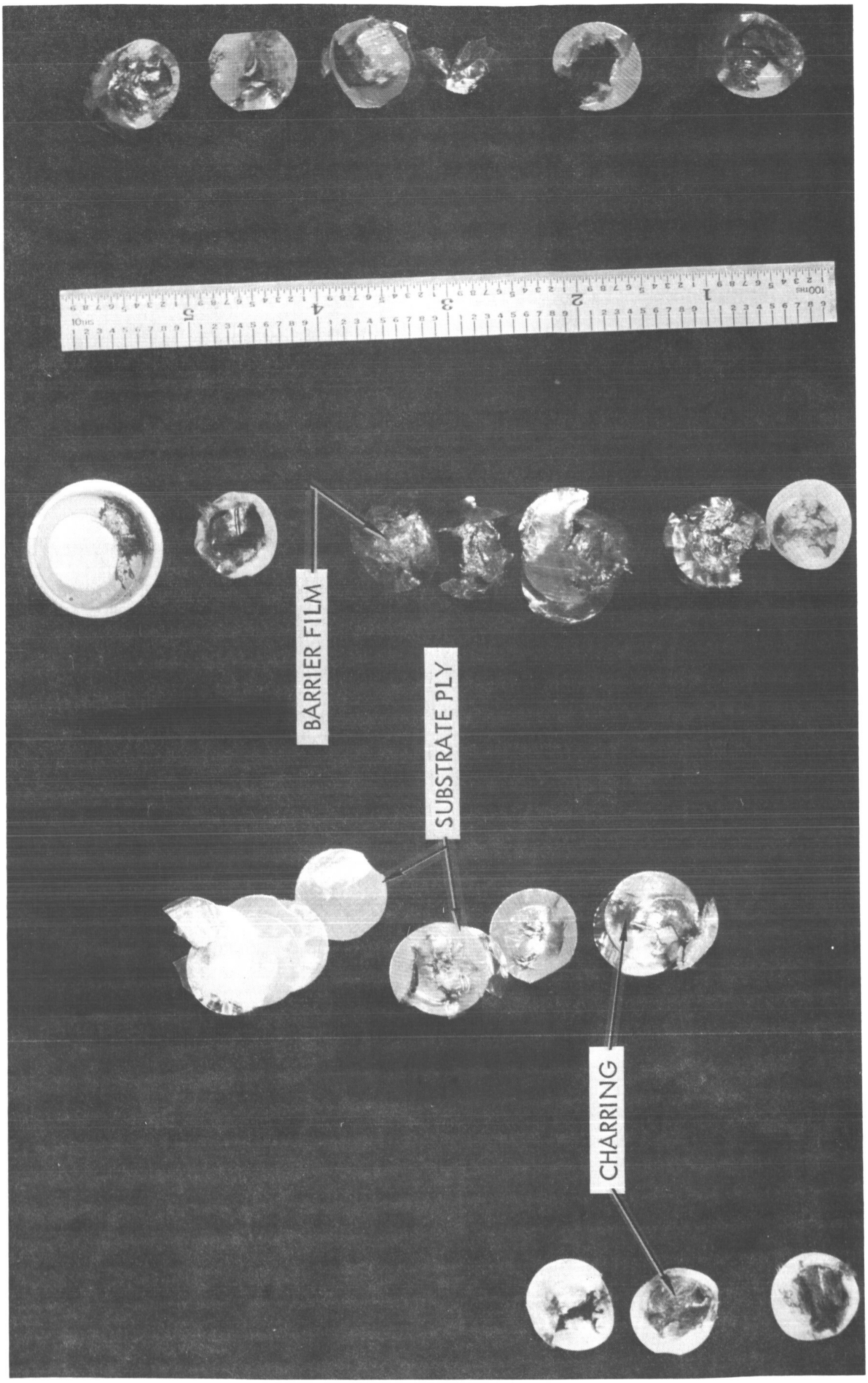

FIGURE 17 - TYPICAL FILM SAMPLES AFTER TEST

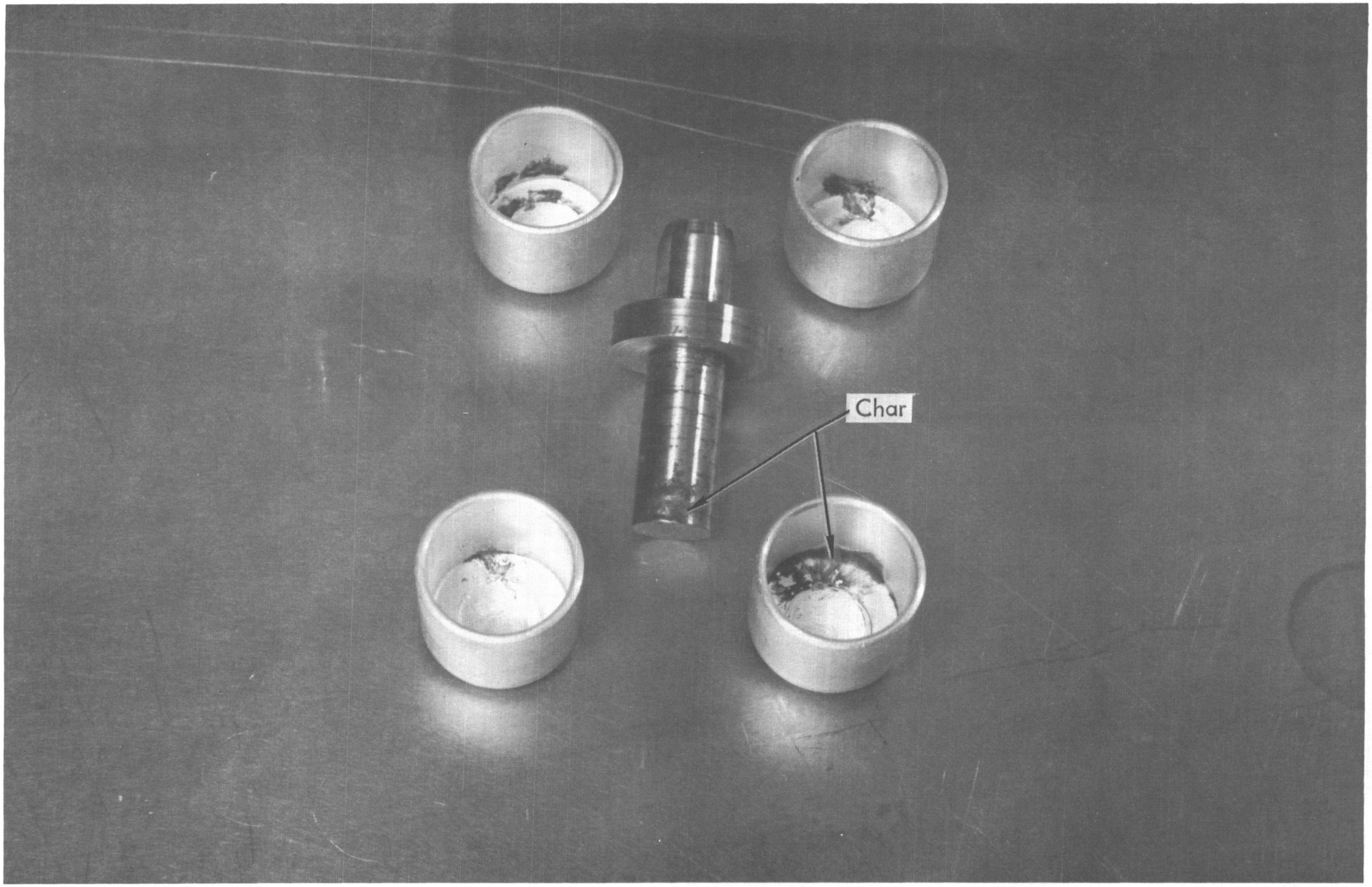

FIGURE **18** - PIN AND CUPS AFTER TEST

5.0 CONCLUSIONS

1. None of the seamed polymeric film specimens successfully passed the ABMA LOX compatibility requirement of 72 ft-lbs. However, tests confirmed that plain Kapton, FEP/Kapton and FEP/Kapton/FEP are LOX compatible and that with the advent of a suitable adhesive system and refined fabrication procedures LOX compatible polymeric systems can be developed. The latter point was demonstrated with the press laminated FEP/Kapton-to-FEP/Kapton/FEP samples.

2. Testing multi-ply film samples, per the ABMA LOX impact test, produces detrimental side effects which gives indicated energy threshold levels below that of a single film ply. No beneficial "cushioning" effect was experienced.

3. Long exposure of Mylar and Kapton film (with an adhesive seam) to liquid oxygen, increases their impact sensitivity and degree of reactivity. The impact threshold level decreases approximately 1 Kg-m (7 ft-lbs) and the reactivity progresses from a mere char to the explosive state. The exposed samples were tested in a LOX saturated condition.

4. Multi-ply sample testing, using the methods and techniques established under the existing ABMA LOX impact test, is practical, and usable data can be obtained. The data does not appear to correlate with single-ply data; however, more extensive tests are required before a firm conclusion can be drawn.

   Multi-ply testing does give a lower indicated threshold level for a material than does the single-ply test. As a result, materials acceptable under current ABMA standards may not be acceptable if tested in multi-ply form. This could be significant if the intended use of that material requires a multi-layer construction.

6.0 RECOMMENDATIONS

1. Additional LOX impact sensitivity testing should be performed on multi-ply samples to determine if a correlation between single-ply and multi-ply test results can be made. The study should include an analysis of the nature and magnitude of the boundary layer forces which are generated under the high impact loadings within the multi-ply samples.

2. Since the initiation of this program several polyimide adhesive systems have been developed which are compatible with existing expulsion bladder fabrication techniques. The use of such a system as a replacement for the polyester adhesive system, GT-300, should increase the LOX impact insensitivity level of a bonded Kapton film composite. Selective polyimide adhesive systems should therefore be investigated for LOX bladder usage.

3. The ABMA LOX Impact Sensitivity Test Specification should be revised to include a provision for multiple-ply sample testing. It appears evident that a new standard of acceptance would have to be established for multiple ply specimens. Such a standard would have to be established only after a large number of specimens constituting a variety of materials had been evaluated. Also, the investigation mentioned in Item 1 above should be completed prior to the establishment of such a standard.

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